Customer No. 30223 **PATENT**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application Of:	)	Atty. Docket No.: 47168-00035USC1
	)	
Richard J. Lazzara	)	Examiner: Paul Prebilic
Thomas S. Heylmun	)	
Keith D. Beaty	)	Group Art Unit: 3738
	)	
Application No.: 09/237,605	)	
	)	
Filed: January 25, 1999	)	
	)	
For: Infection-Blocking Dental Implant	)	

DECLARATION OF PRABHU GUBBI

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Prabhu Gubbi, declare that:

A. I reside at 4445 SW Oakhaven Lane, Palm City, FL 34990.

B. I have degrees in Mechanical Engineering and Foundry Engineering from Bangalore University, India. I also have a Ph.D. degree in Materials Engineering from Auburn University. Since receiving my Ph.D., I have been employed as an engineer with several corporations where I have worked as a materials scientist. Currently, I am a materials scientist with Implant Innovations, Inc ("***3i***").

C. In the course of my work at ***3i***, I have examined numerous surfaces of objects used in dental implantology. One of the machines that I use to examine these surfaces is a Scanning Electron Microscope (SEM). The machine that I use is made by Aspex Instruments

LLC, Model No. PSEM II. Another machine used to measure surface roughness and surface area is a MicroXAM Surface Mapping Microscope (SMM) made by ADE Phase Shift, Model No. Micro XAM 100.

D. At the request of *3i*'s attorneys, I have carried out certain inspections of commercially pure titanium implants after they have been exposed to various treatments and compared the results with those of the methodology in the subject patent application, which is owned by *3i*. The results of this work are discussed below. In one series of tests (Exhibit A), a titanium dental implant was given a treatment according to the method described in this patent application to produce an Osseotite® surface, which is commercially available on *3i*'s dental implants. In the second series of tests (Exhibit B), titanium implants were exposed to the two-step procedure described in a Japanese published patent application, JP 3146679 A2 to Haruyuki. A third series of tests (Exhibit C) exposed titanium implants to a group of mineral acids. A fourth series of tests (Exhibit D) exposed titanium implants to a grit blasting step, followed by exposure to a group of mineral acids. I understand that my report is to be submitted to the U.S. Patent and Trademark Office in connection with the subject patent application.

First Test - Osseotite® Surface (Exhibit A)

E. A commercially pure titanium dental implant, after machining to form the implant's threaded shape, was first immersed in 8.45 wt% hydrofluoric acid for 60 seconds to remove the native oxide layer. After rinsing in deionized water with baking soda, followed by a rinse in deionized water, the implant was immersed in a mixture of one part by volume of 37 wt% hydrochloric acid and 7.5 parts by volume of 84.5 wt% sulfuric acid for 7 minutes at 60-70°C. The resulting surface is shown in Exhibit A, which includes an SEM photograph having a magnification of 2,000 times taken with the SEM machine and a three-dimensional

representation of the Osseotite® surface produced by the SMM machine adjacent to the SEM photograph. For comparison, the second sheet in Exhibit A includes an SEM photograph of the intermediate surface after using hydrofluoric acid to remove the native oxide (Stage I), and an SEM photograph after that intermediate surface has been further etched by the mixture of hydrochloric and sulfuric acids to achieve the Osseotite® surface (Stage II). As seen in these SEM photographs, the mixture of hydrochloric and sulfuric acids further roughens the intermediate surface after its native oxide was removed with hydrofluoric acid.

Second Test - Japanese Patent Application (Exhibit B)

F. A series of 4 mm diameter dental implants made of commercially pure titanium were taken from regular production after machining to form the implant, but before the implants had been provided with any type of treatment. Except for Comparative Example 1, each implant was dipped in hydrofluoric acid solutions using the conditions in the examples of the translation of the Japanese patent application, JP 3146679 A2, and recorded in Table "B" in the front of Exhibit B. Post-Treatment with mixtures of hydrofluoric acid and hydrogen peroxide was carried out where the examples used such post-treatments.

G. I photographed each implant after it was exposed to the treatments in the examples of the Japanese Patent Application using the SEM machine. SEM photographs having a magnification of 2000 times were taken of each implant and are shown in Exhibit B, identified by the example designations stated in the translation of JP 3146679 A2 at pages 5-6. In addition to the SEM photographs, the SMM machine was also used to examine each implant after the treatments, and the SMM representation of each surface is shown adjacent to the SEM photograph. A region of the surface measuring 162.8 μm by 123.3 μm was examined with the SMM machine. The area of this region would be 20,073 μm^2 if it were a flat plane. The

Comparative Example 1 was used as the base line for the area of the machined surface before pre-treatment and post-treatment. The Comparative Example 2 provides insight on the effect of only the pre-treatment step on the machined surface, since no post-treatment was performed in Comparative Example 2.

H. I conclude from the information provided in Table "B" that the maximum increase in the surface area was only 7.5%, which was found in the Experimental Example 2. From these SEM photographs and the three-dimensional representations of the surfaces, it appears that exposure of titanium implants to hydrofluoric acid treatments produced less roughening than reported by the Japanese Patent Application. In fact, the machining marks are still visible on many of the surfaces. Further, the post-treatment with hydrofluoric acid and hydrogen peroxide appears to smoothen the surface, which is consistent with the teaching at column 2 on page 4 of the translation of the Japanese Patent Application. Finally, the treatments of the Japanese patent application produced surfaces that do not resemble the surface achieved by the methodology of the subject patent application, as shown in Exhibit A.

Third Test - Mineral Acid Exposure (Exhibit C)

I. A series of 4 mm diameter dental implants made of commercially pure titanium were taken from regular production after machining to form the implant, but before the implants had been provided with any type of treatment. Each implant was dipped in a mineral acid solution having the concentration and temperature shown in Table "C," located at the front of Exhibit C.

J. I photographed each implant before and after it was exposed to the acid solution using the SEM machine. SEM photographs having a magnification of 2000 times are attached to Exhibit C. The SMM machine was also used to examine each implant before and after acid

treatment. A region of the surface measuring 162.8 μm by 123.3 μm was examined. The area of this region would be 20,073 μm^2 if it were a flat plane. The acid treatments had only a small effect on the surface area. In some cases, the area increased slightly and, in other cases, the area was reduced slightly. A reduction in surface area may be attributed to smoothing of the machining marks on the surface of the implants. The maximum increase in surface area was found to be 18.8%, produced by exposure to 49% HF for 5 minutes at 24°C (Test Sample 4). In that example, the machining marks are no longer visible and the titanium metal grains can be seen on the surface.

K. I conclude from the information provided in Exhibit C that exposure of machined titanium implants to the mineral acids produced little effect, except for hydrofluoric acid, which produced a surface in which the grain structure could be seen. None of the acids were capable of providing a surface roughness of twice the initial value (*i.e.,* 100%). Furthermore, the mineral acids produced surfaces that do not resemble the surface achieved by the methodology of the subject patent application, as shown in Exhibit A.

Fourth Test - Grit Blast Plus Mineral Acid Exposure (Exhibit D)

L. A second series of 4 mm dental implants made of commercially pure titanium was taken from regular production after machining to form the implant, but before the implants were provided with any further treatment. Each implant was subjected to grit blasting by Biocoat, Inc., using aluminum oxide #60 grit at a pressure of 20-60 psig. Thereafter, each grit blasted implant was dipped in an acid solution having the same concentration and at the same temperature as used in the Third Tests (Exhibit C) mentioned above. After remaining in the acid solution for the length of time stated in Table "D," which is at the front of Exhibit D, each implant was rinsed in reverse osmosis/deionized water, isopropyl alcohol and dried with a hot air gun.

M. I took photographs of each grit-blasted implant before and after it was exposed to the acid solution using the SEM machine. SEM photographs having a magnification of 2000 times are attached to Exhibit D. The SMM machine was also used to examine each implant before and after acid treatment. A region of the surface measuring 162.8 μm by 123.3 μm was examined. The area of this region would be 20,073 μm^2 if it were a flat plane. It is evident that the grit blasting increased the area significantly, compared to the surface of machined implants. The grit-blasted surface area was increased by acid treatment in some cases and decreased in others, with the maximum increase being 10.7% after exposure to nitric acid (Test Sample 1) and the maximum decrease on surface area being 34.4% after exposure to hydrofluoric acid (Test Sample 4). In no case did the acid exposure increase the surface area by a factor of two (*i.e.*, 100%).

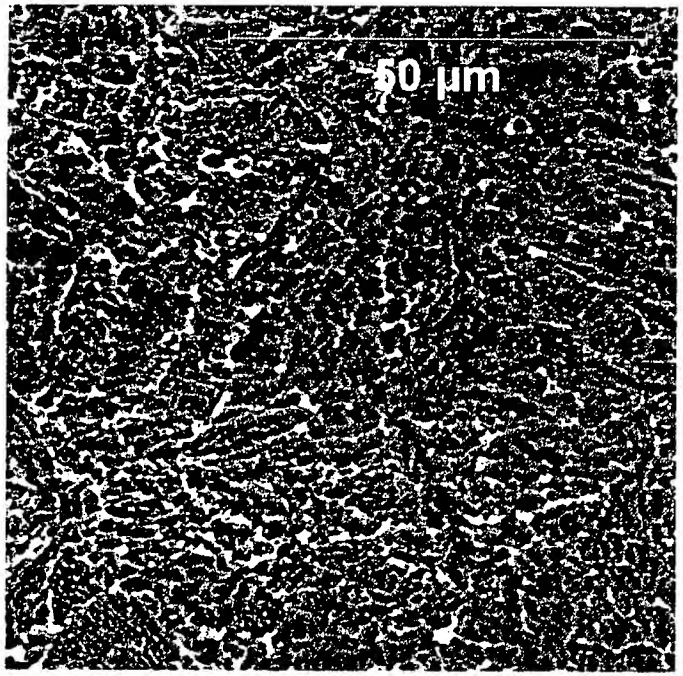
N. I conclude that the exposure of the grit-blasted implant surfaces to these mineral acids did not produce a surface resembling the surface shown in the photographs of the subject patent application, as shown in Exhibit A.

O. The undersigned, being hereby warned that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this Declaration, declares that the facts set forth in this Declaration are true, and all statements made of this own knowledge are true, and all statements made on information and belief are believed to be true.

Date: 05-23-2003

Prabhu Gubbi

Exhibit A: Osseotite SEM and 3-D Surface Map



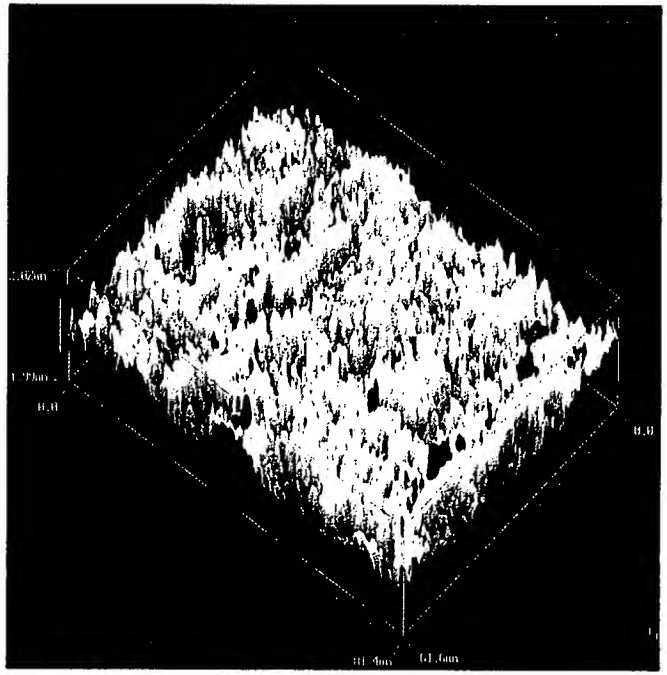
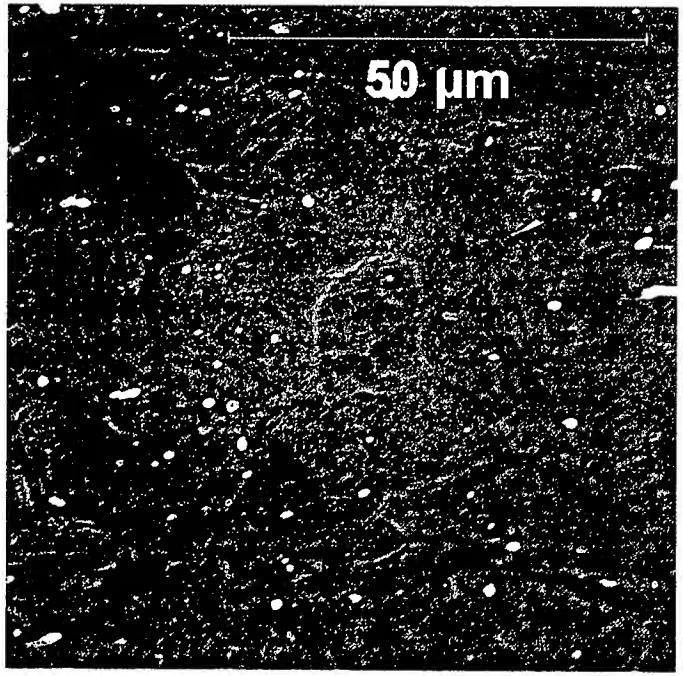
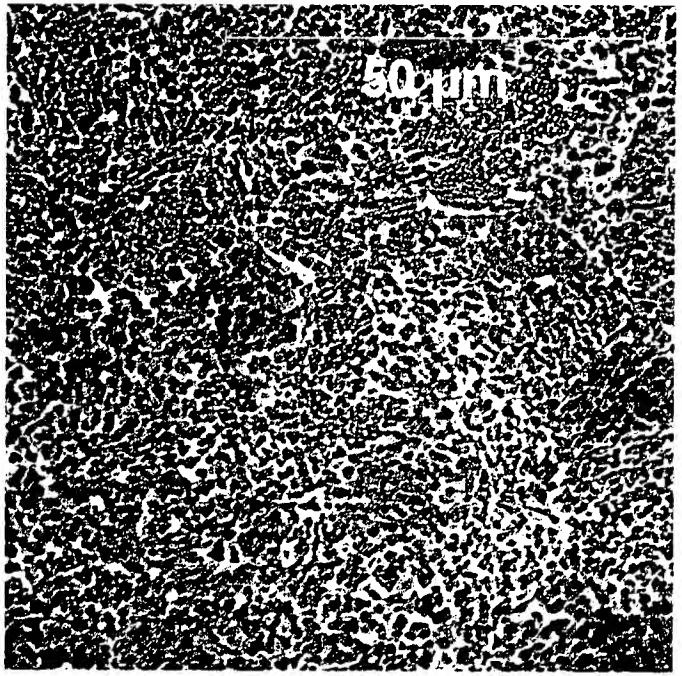


Exhibit A: SEMS after HF, and after Mixture of H_2SO_4 + HCl



Stage I. After Etching in HF



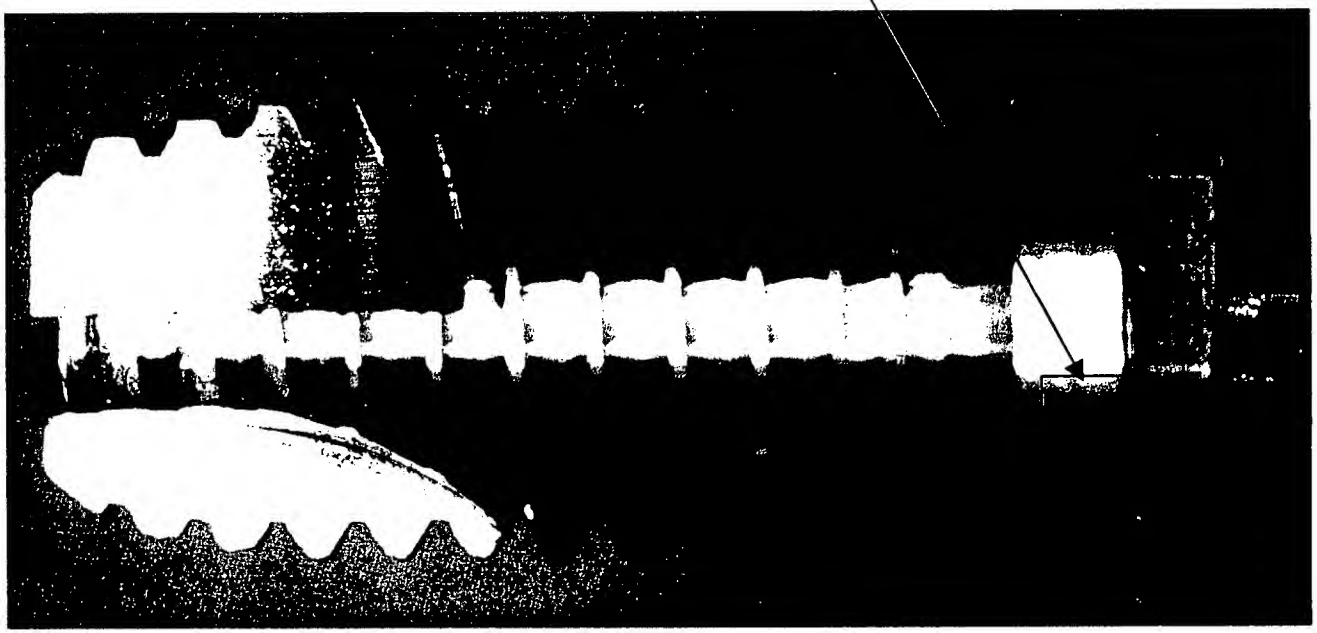
Stage II. After Etching in HF and Etching in H_2SO_4 + HCl

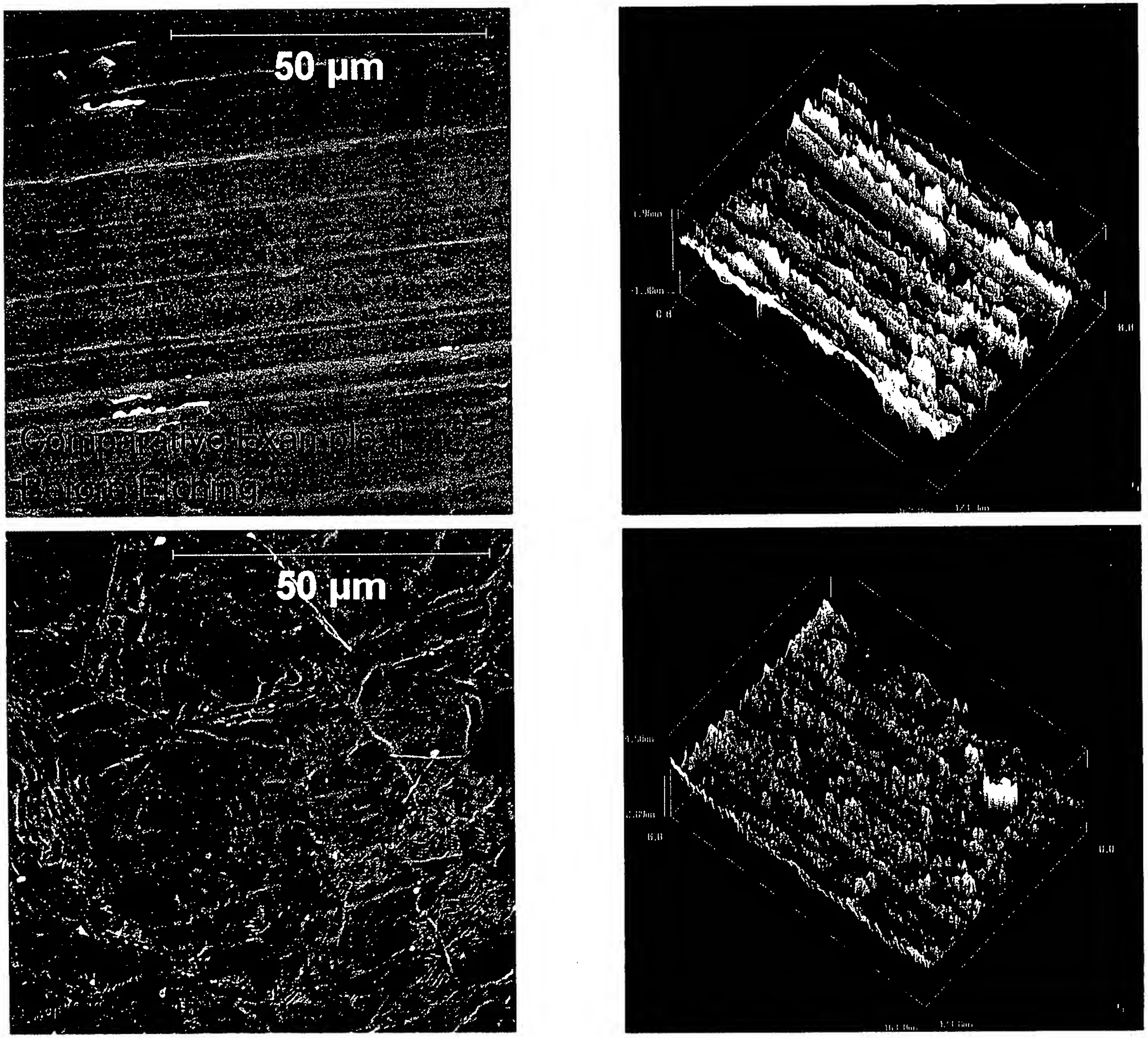
Table B. Results of Tests Conducted According To JP 3146679 A2

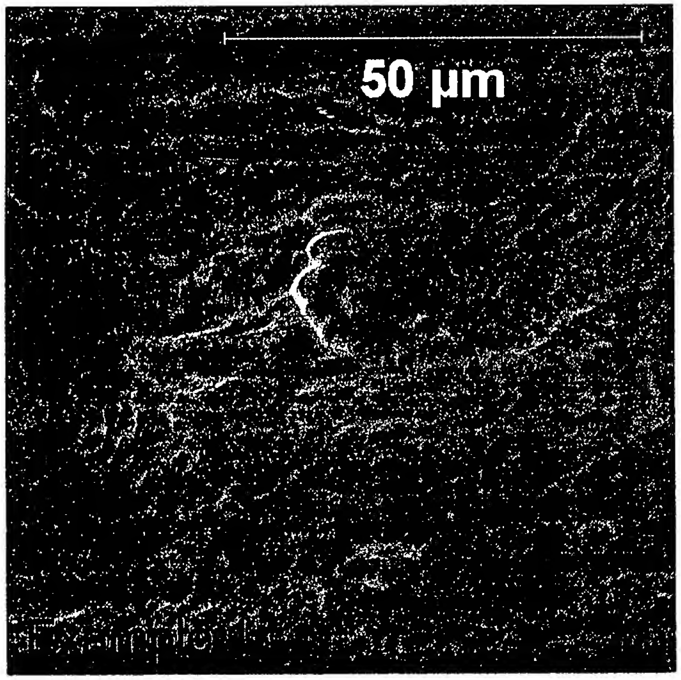
Test Sample No.	Pre-Treatment and Post-Treatment Conditions	Surface Area Before Pre & Post-Treatment (μm^2)	Surface Area After Pre & Post-Treatment* (μm^2)
Comparative Example 1	No Pre-Treatment No Post-Treatment (i.e, Machined Surface)	21110.36	N/A
Comparative Example 2	Pre-Treatment: 4% HF, 1 min., No Post-Treatment	21110.36	22660.69* *Surface Area after Pre-Treatment, No Post-Treatment
Example 1	Pre-Treatment: 4% HF, 30 sec. Post-treatment: 4% HF + 8% H2O2, 15 sec.	21110.36	21791.26
Example 2	Pre-Treatment: 4% HF, 1 min. Post-treatment: 4% HF + 8% H2O2, 15 sec.	21110.36	21992.64
Example 3	Pre-Treatment: 4% HF, 2 min. Post-treatment: 4% HF + 8% H2O2, 15 sec.	21110.36	22201.61
Example 4	Pre-Treatment: 2% HF, 1 min. Post-treatment: 4% HF + 8% H2O2, 15 sec.	21110.36	21820.26
Example 5	Pre-Treatment: 8% HF, 1 min. Post-treatment: 4% HF + 8% H2O2, 15 sec.	21110.36	22325.15
Experiment Example 1	Pre-Treatment: 4% HF, 1 min. Post-treatment: 8% H2O2, 1 min.	21110.36	22694.75
Experiment Example 2	Pre-Treatment: 4% HF, 1 min. Post-treatment: 8% H2O2, 15 sec.	21110.36	22148.67

Scanning Electron Microscopy and Surface Mapping Microscopy Investigation on Implants

Area of Interest for All Implants




50 µm
Comparative Example
Before Etching
50 µm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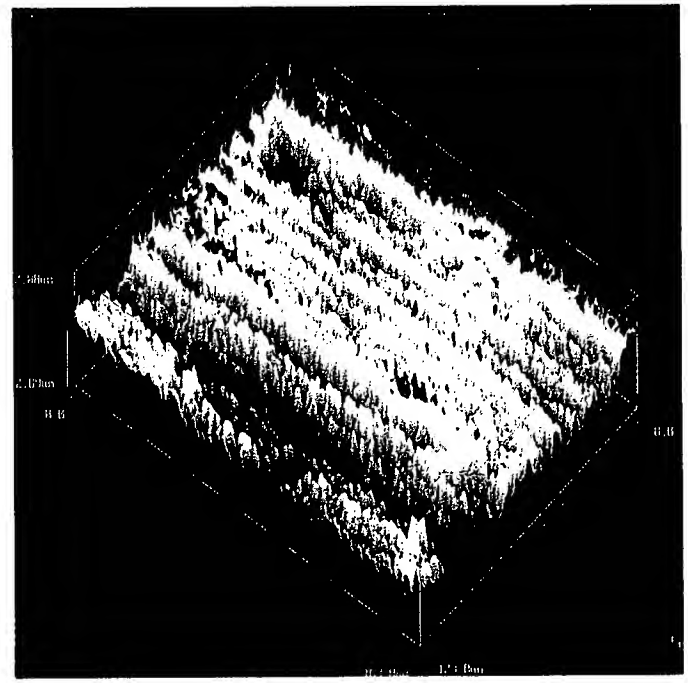

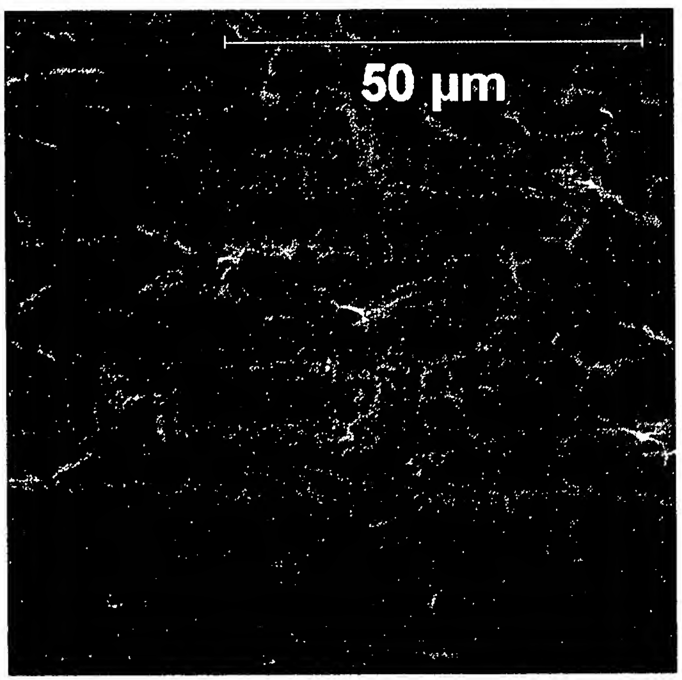
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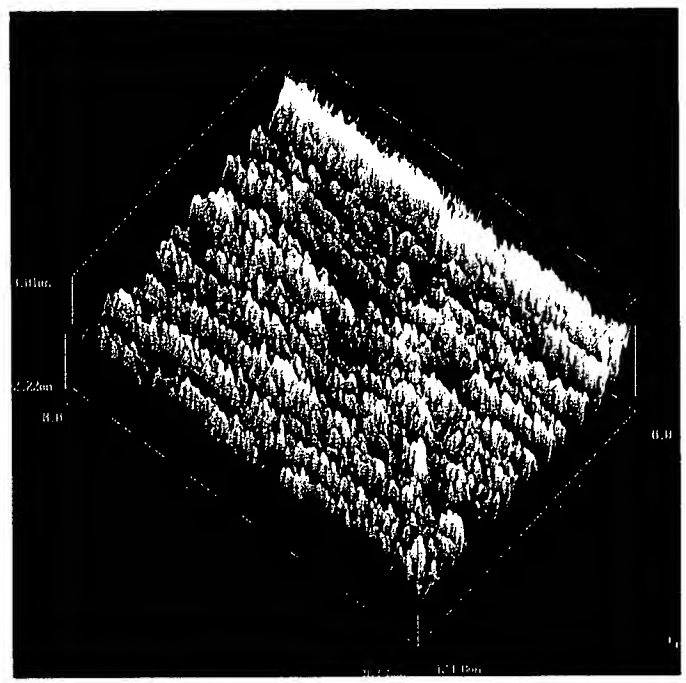
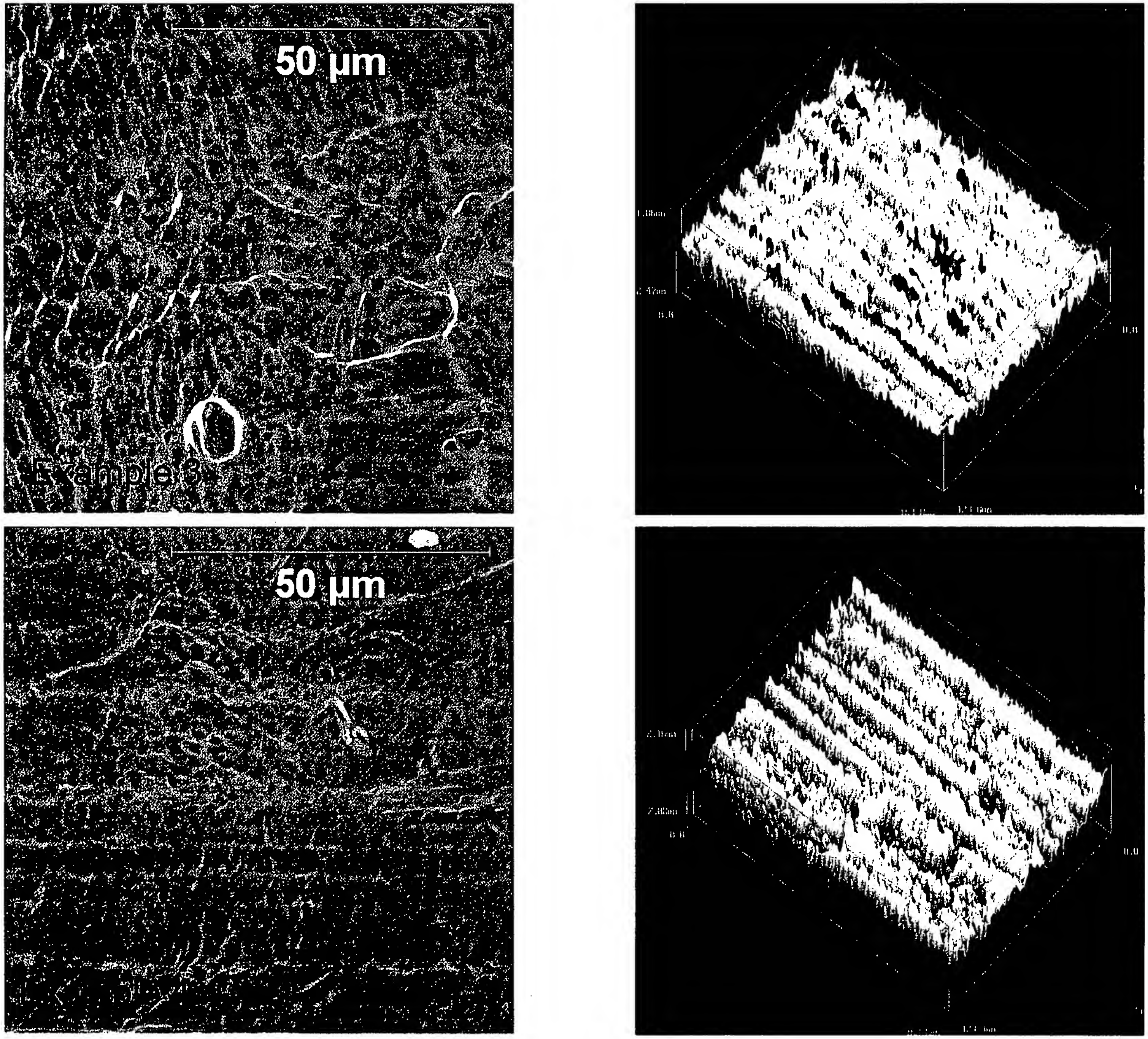
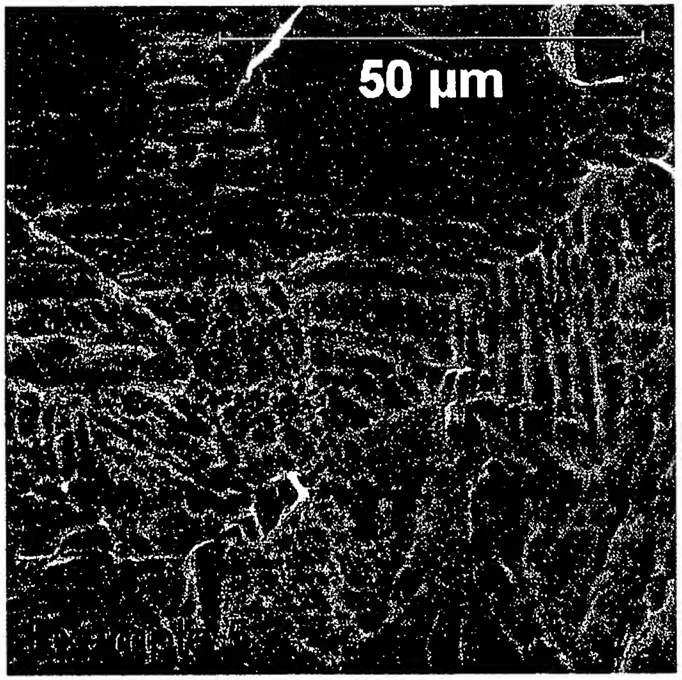



50 µm
Example 3
50 µm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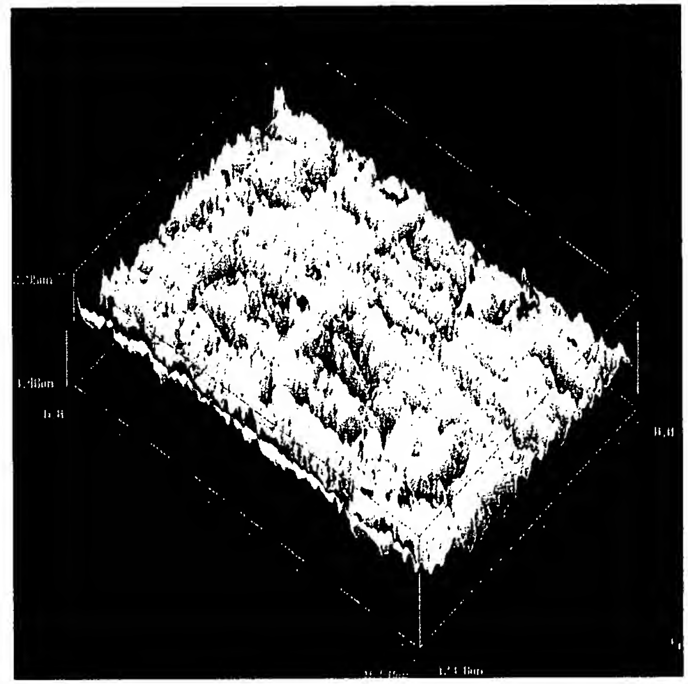
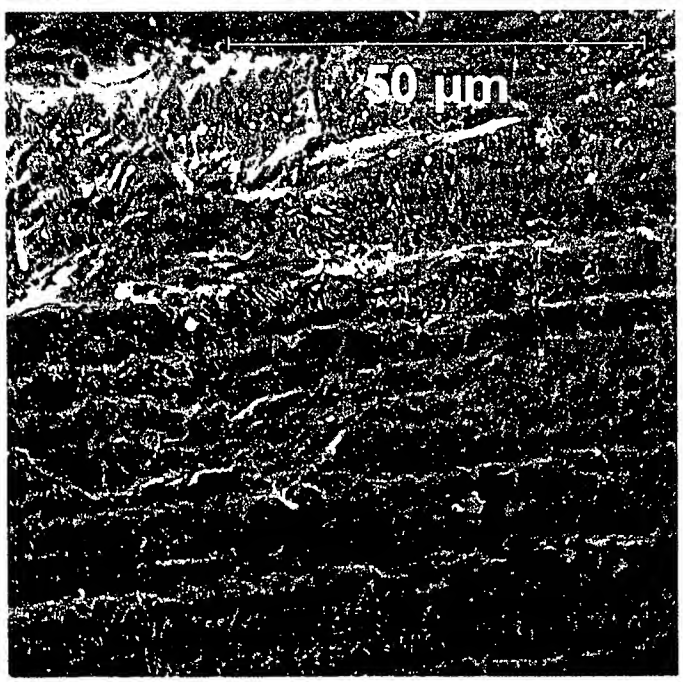
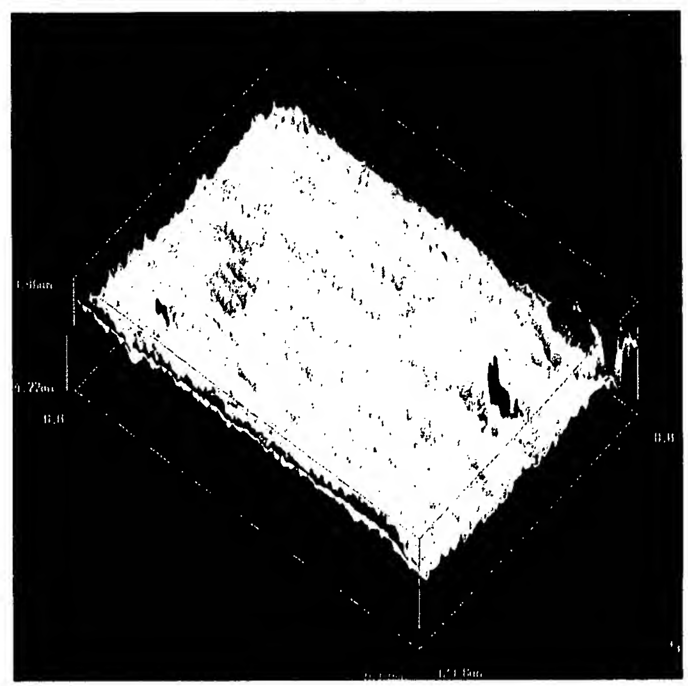
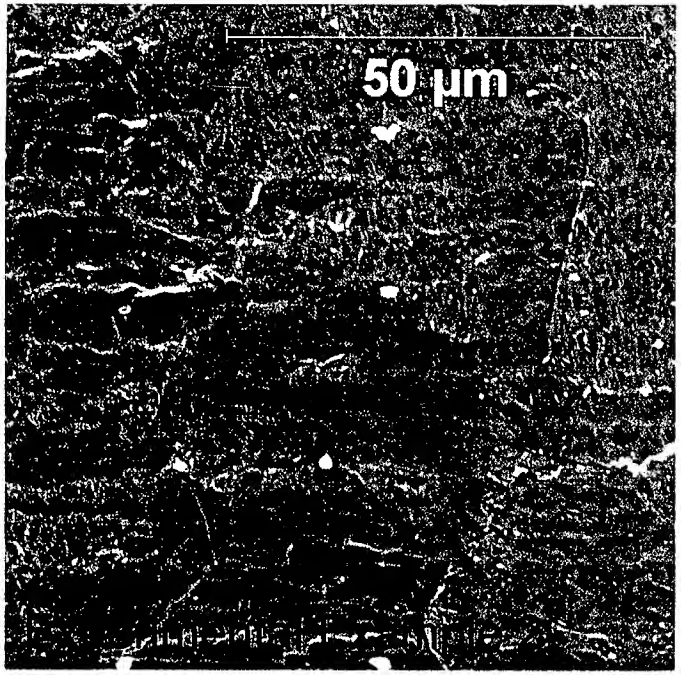



50 µm




50 µm

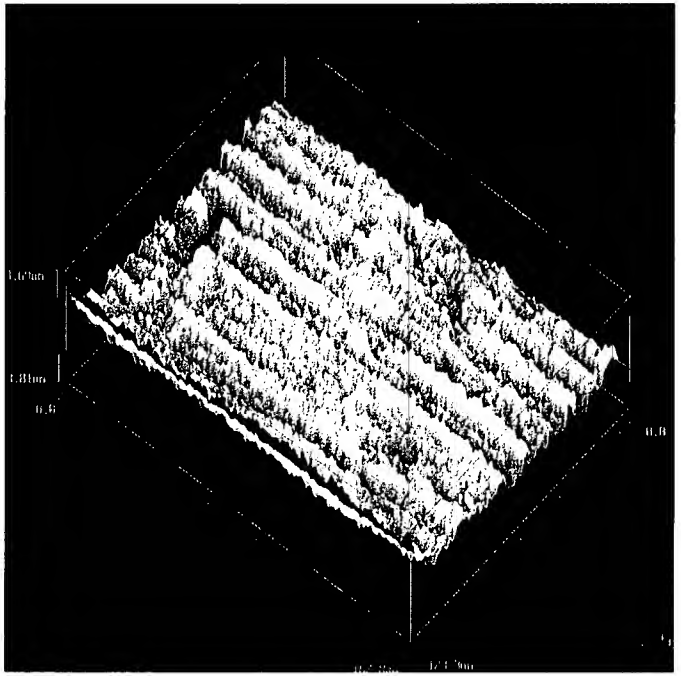


Table C: Results of Etching Machined Surface with Various Min ral Acids

Test Sample No.	Etching Conditions	Surface Area Before Etching (μm^2)	Surface Area After Etching (μm^2)
1	70% Nitric Acid, 5 min. at 24°C	20396.93	20583.66
2	85% Phosphoric Acid, 5 min. at 24°C	20999.55	20314.00
3	96% Sulfuric Acid, 5 min. at 24°C	22019.00	20405.75
4	49% Hydrofluoric Acid, 5 min. at 24°C	20440.97	24289.25
5	37% Hydrochloric Acid, 5 min. at 24°C	20433.19	20274.46
6	70% Nitric Acid, 5 min. at 50-55°C	20845.00	20409.54
7	85% Phosphoric Acid, 5 min. at 50-55°C	20699.07	21270.39
8	96% Sulfuric Acid, 5 min. at 50-55°C	20847.20	20347.88